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Cooperative Charging Effects of Fibers from Electrospinning of Electrically Dissimilar Polymers

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Abstract

Electrical charging and residual charge decay of electrospun nonwoven webs comprised of two electrically dissimilar polymers were studied in an effort to investigate their filtration properties. Polystyrene (PS) and polyacrylonitrile (PAN) were electrospun separately, in a layer-by-layer configuration and simultaneously in a side-by-side bi-component apparatus into thin webs on a polypropylene spunbond substrate. During electrospinning of the PS and PAN polymer solutions, the fibers became positively charged when positive voltage was applied to the solution-filled spinning nozzle and became negatively charged when negative voltage was applied. This study was undertaken to examine the effect of cooperative charging from electrospinning of the two polymers, the effect of the three types of web constructions on charge retention, and filtration properties of the fibers. It was found that single, multilayered, and bi-component webs retained surface charges in the thousands of volts that diminished very little over a 20-hour period, but eventually bled off while resting for three months. Filtration properties were found to be exceptionally high for some, but not all, electrospun samples; filtration was found to have a weak dependence on both surface charge and web geometrical factors, particularly the fiber diameter, that influence pressure drop of the aerosol test.

Key Words

Electrospinning, poly(styrene), poly(acrylonitrile), surface charge potential, aerosol, filtration, surface charges, electrospun fiber web.

Introduction

The method of electrospinning polymer fibers from solution has been described in many patents and research papers since the 1930s (1-6). Although the phenomenon of electrospun fibers ejecting from the surface of a charged polymer solution is still being defined through engineering studies and modeling of the electrospinning process (7-11), practical use has been made of these ultrafine fibers for filtration (12-15). In the Proceedings of INTC 2003, we studied charging effects on target coverage during electrospinning (16). We found that fibers of different polymers would repel each other and would not intermingle into a single region of mixed fibers on a charged collection surface. We also reported the charging of polystyrene nanofibers. Polystyrene (PS), a good electrical insulator, was found to carry a relatively high charge potential of 400 volts after electrospinning, which decayed to about 200 volts after 100 minutes. The charge retention on the electrospun polystyrene was notable. We decided to further investigate the effect of fiber charging and web construction on residual charge decay over time and filtration efficiency of the web over time. The following study examined the charging and filtration effects from three types of web construction designed to produce cooperative charging between dissimilarly charged fibers: single component fibers of PS and polyacrylonitrile (PAN) electrospun separately, bi-component fibers of PS and PAN that were electrospun simultaneously, and individual layers of fibers of PS and PAN of different charge electrospun in subsequent time intervals on the same substrate to give a two-layer or four-layer mat. In order to

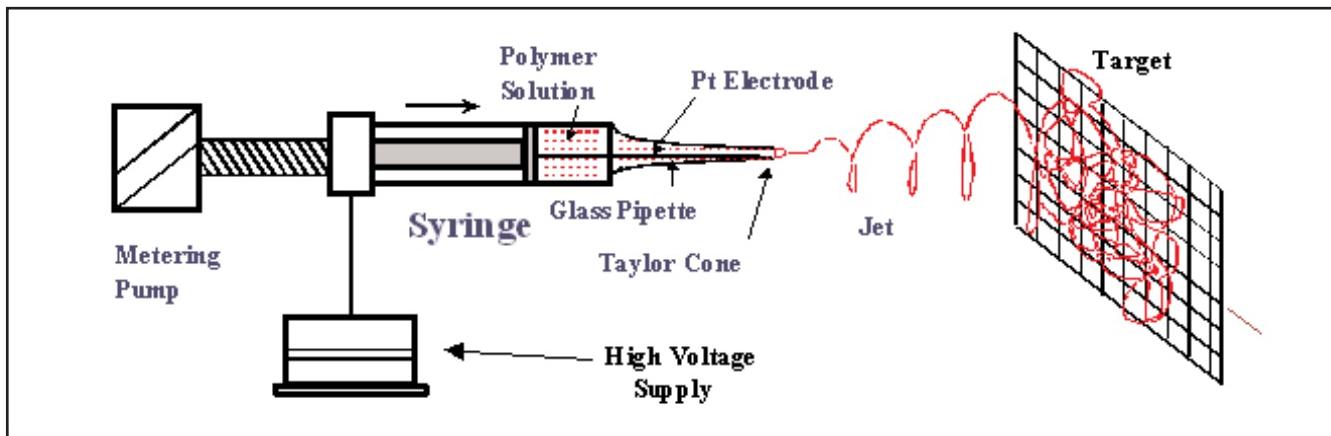


Figure 1
ELECTROSPINNING SETUP FOR LAYER-BY-LAYER SPINNING

accomplish this, it was necessary to use a special spinneret designed for co-spinning fibers to overcome the charge interference between dissimilar fibers that had previously been observed. The bi-component spinning system used in this study had been designed and previously reported to produce intermingled electrospun fibers of two different polymers into a single web (17). The overall objective of this work was to determine whether a bi-component fiber web containing fibers of identical charge polarity would produce a cooperative charging effect causing retention of electrical charge over a long period of time, as in the case of triboelectrically charged fabrics, or whether a multilayered mat of oppositely charged fiber layers would provide extended electrical charge retention for high filtration efficiency. Two polymers were selected as electrically dissimilar materials for this experiment: modacrylic is reported to assume negative charge when carded in triboelectric filter applications (18), so we used an acrylic polymer, PAN, for our study. Poly(styrene), or PS, is a polymer with high electrical resistivity that can hold onto either positive or negative electrical charge for hours after charging (16).

Experimental

Electrospinning Process: A microsyringe pump from the Orion Company was used to control the throughput of the polymer solution. Stainless steel plates (46×61 cm) were used as fiber collectors. Uncharged spunbonded polypropylene fabric was adhesively attached to the metallic collector plate in order to collect the electrospun fibers onto a substrate fabric that was easy to handle for testing. Plastic syringes fitted with metal needles were used as the polymer solution reservoir and nozzle for electrospinning. Power supplies (manufactured by SIMCO) of positive and negative polarities with 50 kV and 2

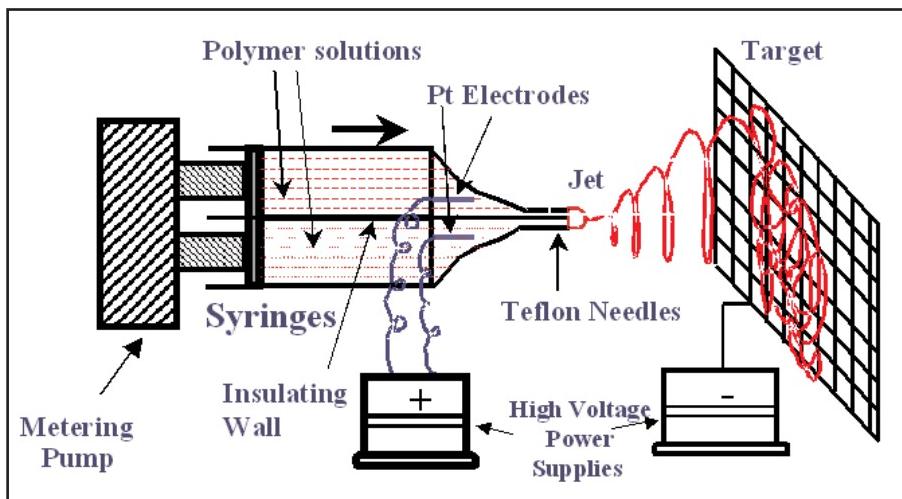


Figure 1
BI-COMPONENT ELECTROSPINNING SETUP

mA were employed as the voltage source for the electrospinning process. One electrode from the power supply was used to charge the polymer solution in the syringe. An electrode from the second power supply was attached to the metal collector plate, held 44 cm from the charged 20-gauge syringe tip. Schematics of both the single reservoir and dual reservoir electrospinning setups are depicted in Figs. 1 and 2.

Solutions of PAN, and PS were electrospun from syringes mounted on a syringe pump (Orion Co.) providing a flow rate of 0.057 ml/min. PAN was supplied by Aldrich in powder form (Mw 135,000) and dissolved in dimethyl formamide (DMF) to a concentration of 10 wt%. PS (Aldrich, Mw 180,000) was dissolved in a mixture of methylene chloride/DMF (90/10 by wt) at 15 wt%. In all the layered configurations (whether single or multi), the PAN solution was electrospun from a syringe needle charged to +15 kV, whereas the PS solution was spun at -15 kV while keeping the collector at -15 kV and +15 kV respectively to maintain an overall potential drop of 30 kV. Bi-component spinning was performed at +18 kV in one case and -18 kV in another case, while keeping the collector at -12 kV and +12 kV respectively to maintain the overall

Table 1
CONSTRUCTION OF ELECTROSPUN FABRICS

Fabric Construction (B/M/M/T)*	Voltage on Solution (kV)	Voltage on Target (kV)	Spin Time per Layer (min)
PS(-)	-15	+15	20
PAN(+)	+15	-15	20
PS(-)/PAN(+)	-15/+15	+15/-15	10 each
PAN(+)/PS(-)	+15/-15	-15/+15	10 each
PS(-)/PAN(+)/PS(-)/PAN(+)	-15/+15/-15/+15	+15/-15/+15/-15	5 each
PAN(+)/PS(-)/PAN(+)/PS(-)	+15/-15/+15/-15	-15/+15/-15/+15	5 each
PS/PAN (+): Bi-component	+18	-12	10**
PS/PAN (-): Bi-component	-18	+12	10**

*(Bottom/Middle/Middle/Top layers. Applied voltage polarity for each layer is shown in parentheses)

** Bi-component electrospinning was conducted at twice the flow rate of the layered samples, flow rate = 0.114 ml/min.

potential drop of 30kV (same average electric field strength as that maintained for the single and multi layered electrospun configurations). The collector, covered with a spunbonded fabric, was charged with an equal charge but opposite polarity of the spinneret.

Fabrics of electrospun fibers applied to a spunbonded polypropylene (PP) were prepared in three different configurations: 1) single component fabrics of PAN and PS; 2) layer-by-layer fabrics of PAN and PS; and 3) bi-component fabrics with blended PAN and PS fibers intermingled within the fabric. *Table 1* shows the configurations and the applied voltages delivered for each system. The PP layer was the bottom layer, followed by sequential layers of electrospun fibers. As shown in *Table 1*, PAN(+) refers to a positive PAN fiber layer on top of the PP, that was electrospun at a positive potential on the polymer solution. Likewise, PAN(+)/PS(-) designates a bottom layer of positive PAN fibers with a top layer of negative PS fibers. Four-layered fabrics are shown with the label, PS(-)

) / PAN(+) / PS(-) / PAN(+), designating PS on the bottom and PAN on the top of the multilayered mat. Surface charge potential measurements were made on the top, or the final layer, of charged fibers.

Measurements: Surface charge potential and filtration efficiency were measured for each sample. A capacitive probe with aperture of 0.07 inch attached to a scanning table was interfaced with an A/D board to measure the surface charge potential at 36 points across 100 cm² of the electrospun fabric (electrospun fibers collected on top of the spunbond polypropylene) held on the metal collector plate. After residual surface charge potential in volts was measured, the fabric was removed from

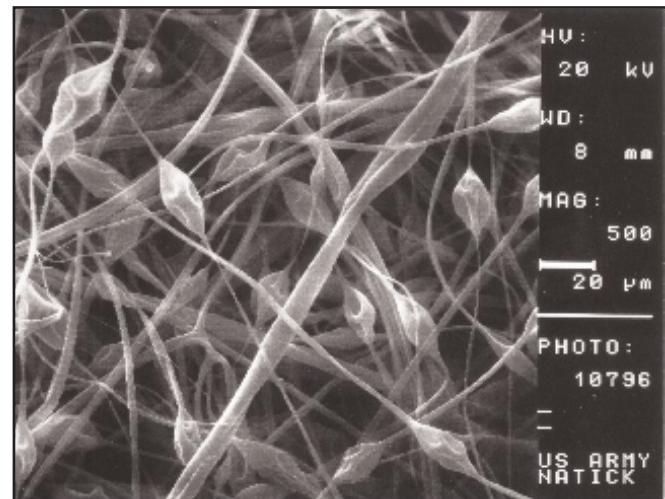
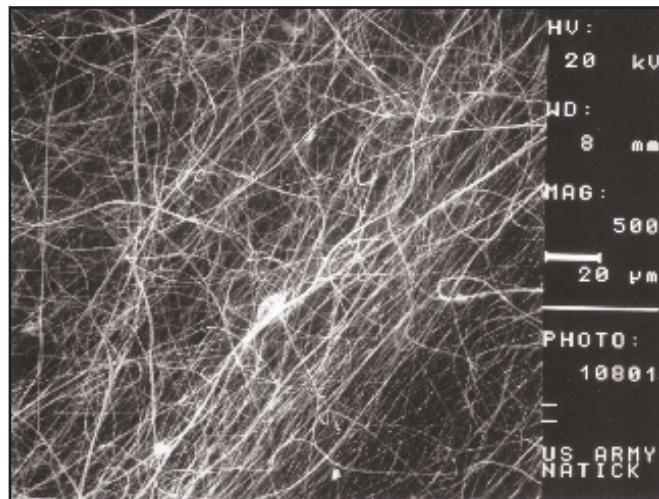
the plate and tested for aerosol penetration. An aerosol generator and detector, the TSI 8130 was used to measure NaCl filtration efficiency (FE) of the electrospun fabrics. Aerosolized NaCl particles had a number average diameter of 0.067 μm with geometric standard deviation of 1.6. The filtration velocity was 5.3 cm/s.

Results

In order to compare charging and filtration results among these samples, total layer thickness was held constant through the use of flow rates and spin times for each layer, as noted above. PAN fibers (ca. 300-700 μm in diameter) were found to be significantly smaller than PS fibers (ca. 1-5 μm in diameter), as seen in the micrographs of *Figure 3*. Bi-component PS/PAN fibers are seen in *Figure 4*, lying across the larger 20 μm spunbond PP fibers

Very high initial surface charge potentials were measured for all electrospun constructions, as seen in *Table 2*. Charge reten-

Figure 3
ELECTROSPUN (A) PAN AND (B) PS FIBERS SPUN ALONE



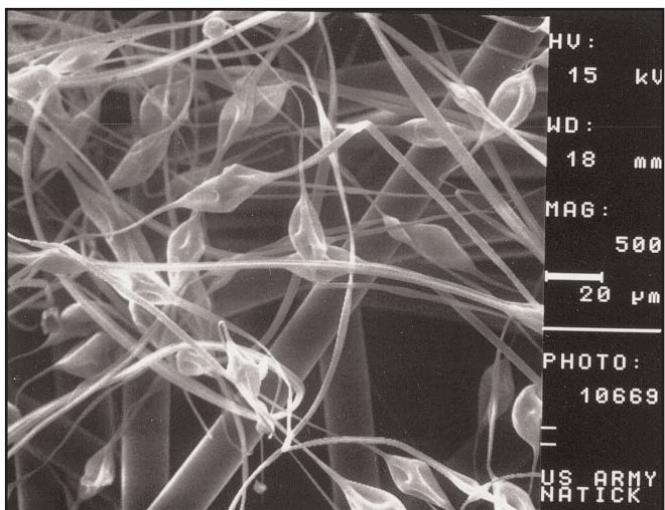


Figure 4
BI-COMPONENT PAN/PS FIBERS
ELECTROSPUN ONTO PP SPUNBOND
SUBSTRATE

tion was measured at 20 hours and after three months of storage in ambient room air. There are no obvious trends; negatively charged surfaces did not retain more charge than positively charged surfaces. Increasing the number of charged layers (in a multilayered construction) did not balance the charge or appear to stabilize the charge over time. In fact, multiple layers and bi-component fabrics seemed to lose charge as much as single fiber layers. However, the samples containing two layers of either PS(+)/PAN(-) or PAN(+)/PS(-) showed an ability to retain charge over a 20-hour period. Over the three-month rest period, most samples lost all residual surface charges (values below 50 volts are not significant). However, three samples showed unusual charge retention over this extended hold period: PAN (+), PS/PAN(+) and PS-co-PAN(-). This set of highly charge stable samples does not suggest any trend or characteristic regarding the polarity of the surface charges, the composition of the web, or the construction of the web as factors in the unusual stability of surface charges in these three cases.

The bar graphs in Figures 5 and 6 depict the charge loss of all electrospun samples over time. There is a significant variance of up to 60% in the surface charge potential over the area of the fibers measured. This variability allows the interpretation of only very rough trends in these figures. We see from the graphs that the bi-component sample that had

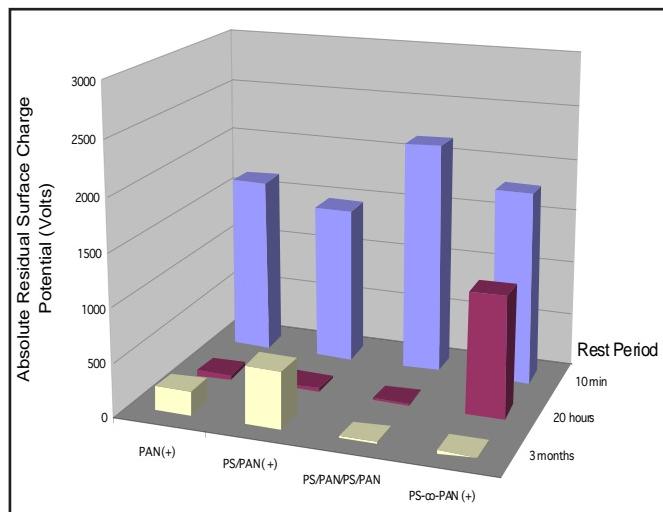


Figure 5
EFFECT OF REST TIME ON RESIDUAL SURFACE CHARGE POTENTIAL FOR SAMPLES OF POSITIVELY CHARGED SAMPLES

been spun from a negatively charged solution retained a surface charge potential of nearly -200 V after three months. This was not the case for the same sample electrospun from a positively charged solution. However, this does not mean that net negative charges are stable. In the case of the one-layer PAN(+) and the two-layer system of PS/PAN(+), we find that the positively charged one-layer and the positively charged two-layer systems retain very high surface charge potential after three months. The negative bi-component PS-co-PAN(-) system retained 14% of its charges; the positive PAN(+) web retained 11%, and the positive bi-layer PS/PAN(+) system retained 25% of its charges. All other samples lost more than 90% of their original surface charges over three months.

Aerosol penetration values were measured after each rest period to determine whether there was an effect of residual charges upon the penetration levels of NaCl. The aerosol test

Table 2
RESIDUAL SURFACE CHARGE POTENTIAL ON ELECTROSPUN SAMPLES

Sample	Surface Voltage after Rest Period (V)		
	10 min	20 hours	3 months
PAN (+)	1950 +/- 804	1427 +/- 477	214 +/- 71
PS (-)	-3332 +/- 804	-2098 +/- 83	-40 +/- 48
PS(-)/PAN(+)	2042 +/- 771	2082 +/- 425	528 +/- 203
PAN(+)/PS(-)	-1784 +/- 475	-1526 +/- 241	16 +/- 2
PS(-)/PAN(+)/PS(-)/PAN(+)	2414 +/- 478	1290 +/- 70	19 +/- 3
PAN(+)/PS(-)/PAN(+)/PS(-)	-1587 +/- 441	-1150 +/- 122	20 +/- 48
PS-co-PAN (+)	1902 +/- 690	1122 +/- 275	24 +/- 48
PS-co-PAN (-)	-1422 +/- 838	-1039 +/- 227	-197 +/- 48

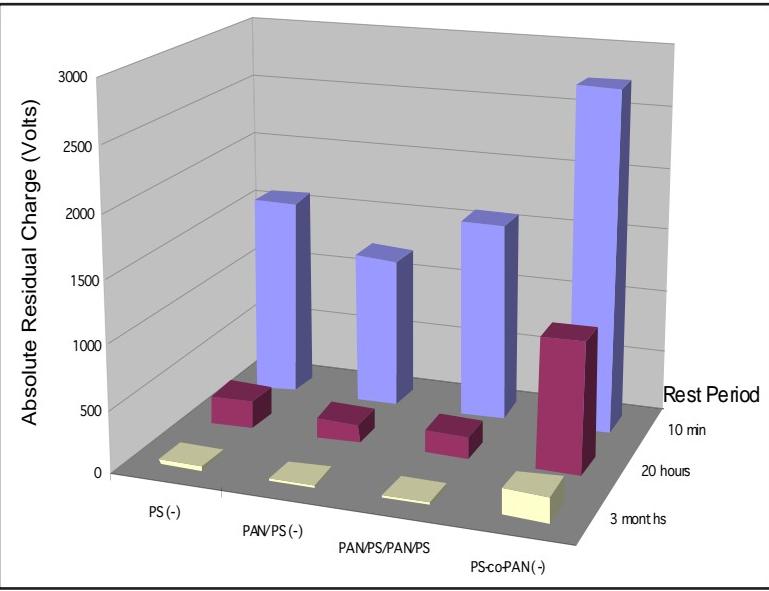


Figure 6
VARIATION OF THE WIDTH OF THE BEST-FIT
RECTANGLE WITH STANDARD DEVIATION OF
ORIENTATION AND AREA OF PORE

Table 3
SURFACE CHARGE POTENTIAL FOR AEROSOL TEST SAMPLES

Sample	Surface Charge Potential after Rest Period (Volts)		
	10 min	20 hours	3 months
PAN (+)	1657	-46	214
PS (-)	-1602	-229	-40
PS(-)/PAN(+)	1463	-33	528
PAN(+)/PS(-)	-1212	-164	16
PS(-)/PAN(+)/PS(-)/PAN(+)	2165	-13	19
PAN(+)/PS(-)/PAN(+)/PS(-)	-1587	-183	20
PS-co-PAN (+)	1801	1122	24
PS-co-PAN (-)	-2743	-1039	-197

Table 4
AEROSOL PENETRATION OF ELECTROSPUN SAMPLES

Sample	% Penetration after Rest Period		
	10 min	20 hours	3 months
PAN (+)	0.14	0.53	4.83
PS (-)	12	15	10.3
PS(-)/PAN(+)	1.2	1.7	3.10
PAN(+)/PS(-)	1.3	4.2	0.909
PS(-)/PAN(+)/PS(-)/PAN(+)	4.2	6.7	1.95
PAN(+)/PS(-)/PAN(+)/PS(-)	0.07	1.74	1.74
PS-co-PAN (+)	0.42	-	12.6
PS-co-PAN (-)	0.15	-	3.09

destroys the sample, so a set of samples different from *Table 2*, but electrospun at the same conditions as outlined in *Table 1*, were used for aerosol testing at 10 minutes and 20 hours after the samples were formed. Samples in *Table 2* were left intact on the collector plate for the entire three-month rest period. Samples used for aerosol tests were removed from the plates and their measured surface charge potential has been clearly affected by handling, as seen when comparing 10 min and 20 hour data in *Table 2* vs. *Table 3*. Aerosol penetration (*Table 4*) and corresponding surfaces charge potential (*Table 3*) are shown below.

Aerosol data are also summarized graphically in *Figure 7*. There is an apparent trend in the data showing that high surface charge potential on electrospun fibers generally causes lower aerosol penetration, although a few data points lie significantly outside the trend shown in *Figure 7*. Further studies need to be performed to confirm this behavior of the weak dependence of aerosol penetration on the charged electrospun fibers.

Another important factor in aerosol penetration of nonwovens is fiber diameter. Thicker fibers produce a web with less fiber surface area that can be penetrated more efficiently by aerosol particles. Larger fiber diameters also result in lower pressure drops across the fabric during aerosol testing at constant flow rate. Data in this study, shown in *Figure 8*, confirm a weak but general trend of increasing aerosol penetration with increasing pressure drop. In addition, when comparing the aerosol penetration of single layer PS and PAN, it can be seen that at all time intervals when the measurements were performed (10 min, 20 hours and 3 months), the aerosol penetration of PS is significantly higher than that of PAN (*Table 4*). Recall that the diameter of PAN fibers (ca. 300-700 nm) was found to be significantly smaller than PS fibers (ca. 1-5 μ m). This suggests that web geometrical factors, in particular the fiber diameter, influence the pressure drop across the electrospun fabrics.

Data in *Figures 7* and *8* demon-

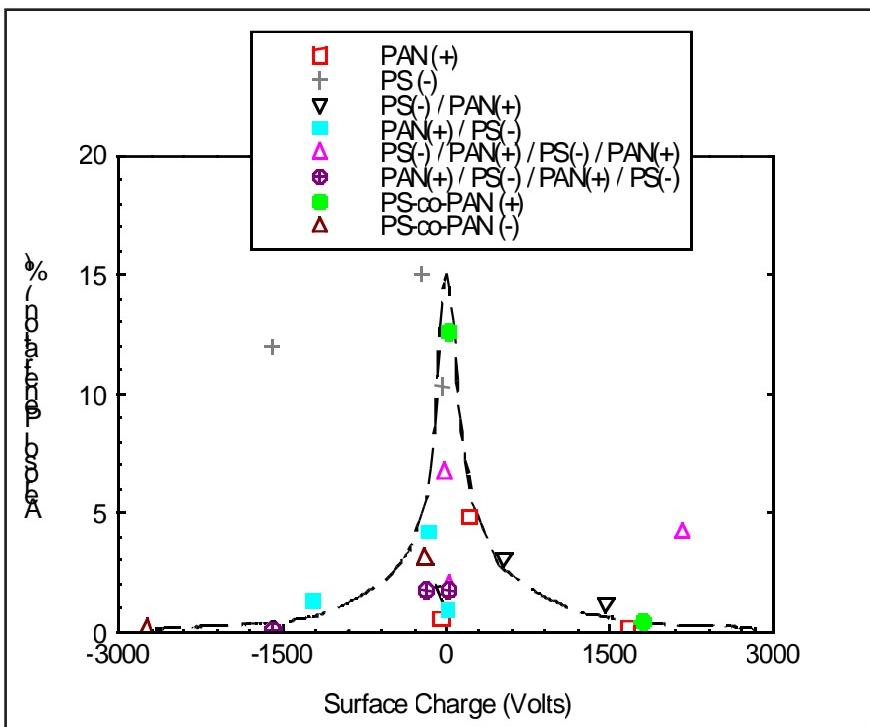


Figure 7

EFFECT OF POSITIVE AND NEGATIVE RESIDUAL SURFACE CHARGE POTENTIAL ON AEROSOL PENETRATION OF ELECTROSPUN FIBERS ON PP SPUNBOND

strate a possible combined effect of fiber surface charges and pressure drop upon filtration efficiency for electrospun fibers; a combined influence that is not apparent for other nonwovens. Although for traditional nonwovens, electrical charges on fabrics have been used to increase filtration efficiency with good charge retention for certain types of charging conditions including triboelectric charging of electrically dissimilar fibers and charge embedment (electret formation) in fibers (19,20), the present research on finer fiber nonwovens produced by electrospinning suggests a weak effect of charging on filtration efficiency. For melt blown fabrics, it has been shown that electrical charges will dominate the filtration behavior of charged fabrics and overcome geometrical effects, such as fabric basis weight (20). Electrospun fabrics possess extremely fine fibers (10-100x smaller than melt blown fibers); as a result these electrospun fabrics have high pressure drops and high aerosol filtration efficiencies that are not substantially influenced by charge retention on the final fabric; geometrical factors such as fiber size are not overcome by fiber charge levels.

Conclusion

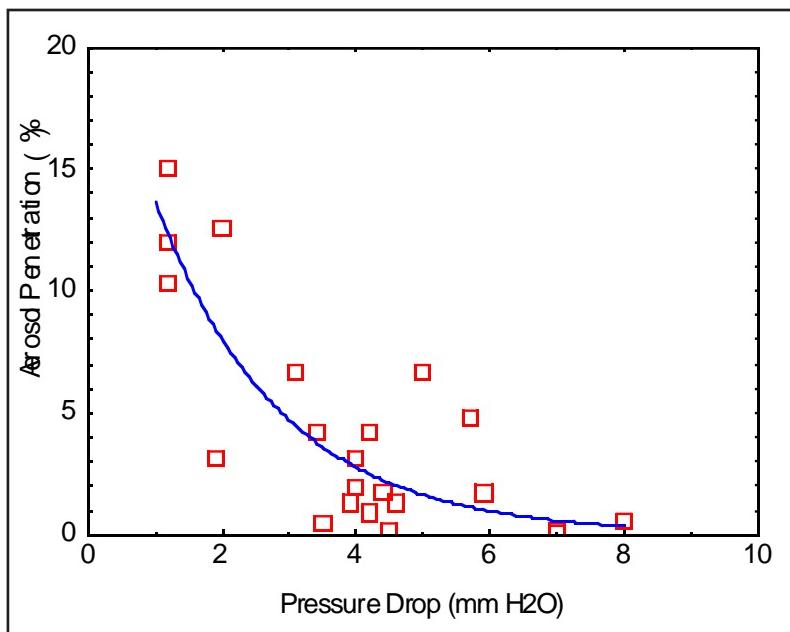
In an effort to investigate the effect of electrical charges on filtration properties and to provide charge stability on the surface of electrospun fibers, we studied the effects of surface charge decay and

web construction of electrically dissimilar fibers in three different configurations on filtration properties. This study demonstrated that extremely high surface charges on the fibers can result from electrospinning, and these charges diminish very little over the course of 20 hours, but are not retained over the course of three months, although three exceptions were found to have significant surface charge potential after this long hold period. In the present investigation involving charged PAN and PS fibers, a weak relationship between electrical charge and aerosol penetration of the web was observed. Although one might expect that electrical charging of the web would overcome the influence of geometrical factors, particularly fiber diameter, on fibers investigated in this study, a dependence of the aerosol penetration on the pressure drop across the web was observed, indicating that fiber diameter influenced filtration in the presence of substantial surface charges. Further studies need to be performed to fully define the dependence of aerosol penetration on the magnitude of charges on electrospun fibers

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Figure 8
EFFECT OF PRESSURE DROP ACROSS THE FABRIC ON AEROSOL PENETRATION ($R^2 = 0.55$)



Performance (MAP), a multidisciplinary university research initiative project. The authors would like to thank Mr. Dave Ziegler and Dr. Joel Carlson for their examination of the fibers by TEM and FTIR.

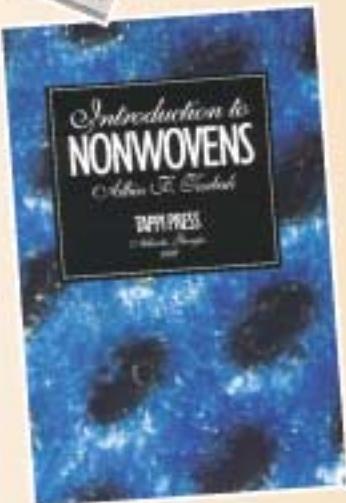
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